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[54]发明名称 离子交换膜燃料电池电极扩散层碳纤维复合纸及其制备方法

[57]摘要

本发明是离子交换膜燃料电池电极扩散层要求的碳纤维复合纸及其制备方法,复合纸由2-10%纤维素浆为基体、80-95%二次活化处理碳纤维和0-10%碳粉组成,制备包括:a.碳纤维或碳粉进行化学活化处理,b.再经过物理活化处理,c.碳纤维长丝切短;d.在分散剂水溶液中分散;e.制得纸浆;f.将碳纤维分散液和浆液按比例混合,加入亲水或疏水表面活性剂,按常规湿法制出复合纸。本发明制成的碳纸,有良好的导电性和透气性,大的比表面积和合理的孔隙度,碳纸两面亲水疏水性能不同,具有定向排水输气功能,克服了目前离子交换膜燃料电池电极扩散层所用的碳纸比表面积较小,碳纸两面亲水疏水性能相同和碳纸柔软性差等不足。

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合纸的制备方法，其特征在于：步骤a一次活化化学处理化学活化剂用量为碳纤维或碳粉重量的10%-50%；化学活化温度为700-900℃；化学活化时间为0.5-1h；化学活化气氛为氮气氛。

8. 根据权利要求 6 所述的离子交换膜燃料电池电极扩散层碳纤维复合纸的制备方法，其特征在于：步骤 b 物理法处理物理法处理温度为700-900℃；时间为 0.5-1h；气氛为水蒸气气氛。

9. 根据权利要求 6 所述的离子交换膜燃料电池电极扩散层碳纤维复合纸的制备方法，其特征在于：步骤 c 的碳纤维长丝切短或磨碎在 1－25mm 范围内，以 3－15mm 为较佳选择。

10. 根据权利要求 6 所述的离子交换膜燃料电池电极扩散层碳纤维复合纸的制备方法，其特征在于：步骤 d 的碳纤维或碳粉在分散剂水溶液中的浓度为 1－6wt％。

碳纤维纸由一层或多层构成.

本发明提供一种离子交换膜燃料电池电极扩散层碳纤维复合纸的制备方法，依次包括下列步骤:

a.将选择的碳纤维或碳粉进行化学活化处理(一次活化处理)，活化剂可选择碱、盐和酸，如氢氧化钠、氢氧化钾、氯化锌、磷酸，活化完毕用蒸馏水将活化剂洗除;

b.将一次活化后的碳纤维或碳粉再经物理活化处理(二次活化处理)，活化过程中选择三氯化铁作催化剂，活化完毕用蒸馏水将催化剂洗除;

c.将过活化后的碳纤维长丝切短或磨碎;

d.配制 0.1% 的分散剂水溶液，然后使活化后的碳纤维或碳粉在分散剂水溶液中分散;

e.将纤维素浆分散，制得叩解度为 18－25° 的纸浆;

f.将碳纤维分散液和浆液按 2: 1－4: 1 比例混合，加入碳纤维分散液 1-5%的碳粉分散液和碳纤维分散液 1%的亲水或疏水表面活性剂，放入搅拌机混合搅拌，既可得到造纸混合浆料;

g.按常规湿法造纸技术将造纸混合浆料制出所需的碳纤维复合纸.

本发明还提供如下方法: 步骤 a 一次活化化学处理化学活化剂用量为碳纤维或碳粉重量的 10%-50%; 化学活化温度为 700-900℃; 化学活化时间为 0.5-1h; 化学活化气氛为氮气氛.

步骤 b 物理法处理物理法处理温度为 700-900℃; 时间为 0.5-1h; 气氛为水蒸气气氛.

步骤 c 的碳纤维长丝切短或磨碎在 1－25mm 范围内，以 3－15mm 为较佳选择.

步骤 d 的碳纤维或碳粉在分散剂水溶液中的浓度为 1－6wt%.

本发明制成的燃料电池扩散层复合碳纸，既有良好的导电性，又有很好的透气性、大的比表面积和合理的孔隙度且制成的复合碳纸两面亲水疏水性能不同，具有定向排水输气功能，同时还具有柔软性. 它克服了目前离子交换膜燃料电池电极扩散层所用的碳纸比表面积较小，碳纸两面亲水疏水性能相同和碳纸柔软性差，不能弯曲等不足.

本发明提供的离子交换膜燃料电池电极扩散层是经过二次活化或经过电化学处理后的活性碳纤维和碳粉为导电相，以天然或人工合成纤维素浆粕为基体，加入适量的分散剂和造孔剂等其他助剂，两次沉浆制成.

液 1-5%的碳粉分散液和碳纤维分散液 1%的亲水或疏水表面活性剂，放入搅拌机混合搅拌，既可得到造纸混合浆料；

g.按常规湿法造纸技术将造纸混合浆料制出所需的碳纤维复合纸。

化学活化法处理实施例之一：将碳纤维或碳粉与其重量 20%的氢氧化钠混合后在 900℃氮气氛中恒温处理 0.5h，然后用蒸馏水将碳纤维或碳粉中的氢氧化钠洗除。

化学活化法处理实施例之二：将碳纤维或碳粉与其重量 30%的氢氧化钾混合后在 800℃氮气氛中恒温处理 0.5h，然后用蒸馏水将碳纤维或碳粉中的氢氧化钾洗除。

物理法活化处理实施例之一：将碳纤维或碳粉与其重量 20%的三氯化铁混合后在 900℃水蒸气气氛中恒温处理 0.5h，然后用蒸馏水将碳纤维或碳粉中的三氯化铁洗除。

物理法活化处理实施例之二：将碳纤维或碳粉与其重量 20%的三氯化铁混合后在 800℃水蒸气气氛中恒温处理 0.5h，然后用蒸馏水将碳纤维或碳粉中的三氯化铁洗除。

本发明制成品具有以下特点：

1. 吸附性能好，比表面大，有较好的电化学性能，适宜作燃料电池电极的扩散层；

2. 加入经过二次活化的碳粉，能改善复合纸的微空结构，有较好的孔隙度和透气性；

3. 碳纤维预处理并长丝短切或磨碎，制备的碳纤维复合纸有较好的强度和柔韧性；

4. 在机械分散后的浆液中先后加入亲油、亲水性不同的分散剂或助剂，经过两次沉浆，抄纸制得的碳纤维复合纸两面具有不同的亲疏水性能，因此具有定向排水输气功能。

5. 所用设备简单，操作方便，易于工业化生产。

将本发明制备的碳纸扩散层和目前通用的碳纸扩散层（德国 SGL 公司生产）组装成离子膜燃料电池进行了对比测试，结果表明本发明制备的碳纸扩散层组装的离子膜燃料电池性能更加优越。

附图为本发明制备的碳纸（a）和 SGL 公司生产的碳纸（b）实验结果比较图.

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[54] The Title of the Invention:

# Carbon Fiber Composite Paper for the Electrode Diffusion Layer

of a Fuel cell with ion exchange membrane and its Preparing Method

[57] Abstract

The present invention is a carbon fiber composite paper required by the electrode diffusion layer of a fuel cell with ion exchange membrane and its preparing method. The composite paper is composed of a 2-10% of cellulose paste as a substrate, 80 - 90% of secondary activation processed carbon fiber and 0-10% of carbon powder. The preparing process includes: a. performing a chemical activation processing on the carbon fiber or carbon powder; b. performing a physical activation processing; c. shortening long carbon fibers; d. dispersing in dispersant aqueous solution; e. producing paper paste; f. proportionally mixing the water dispersion of carbon fiber with the paste, added with hydrophilic or hydrophobic surfactant so as to produce a composite paper by a conventional wet method. The carbon paper produced by the present invention has a good electric conductivity and a good air permeability with great specific surface area and reasonable porosity. One surface of the carbon paper has a hydrophilic feature and the other surface has a hydrophobic feature and the carbon paper has directional water drainage and air transfer functions so that defects of the electrode dispersing layer of the current ion exchange

membrane of the fuel cell, such as low specific surface area, both surfaces having identical hydrophilic feature or hydrophobic feature as well as the low flexibility, can be removed.

We claim:

1. A carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane, characterized in that, the composite paper takes 2 - 10% of cellulose paste as its substrate, 80 - 95% of carbon fiber and 0 - 10% of carbon powder, wherein the carbon fiber and carbon powder are secondarily activation processed.

2. The carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 1, characterized in that, said carbon fiber can be carbon fiber of polyacrylonitrile, carbon fiber derived from asphalt, carbon fiber derived from micellar, active carbon fiber, graphite fiber, vapor phase growing carbon fiber or a carbon fiber substance of their combination.

3. The carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 1, characterized in that, the carbon powder is acetylene black, graphite milk or active carbon, the substrate material can be selected from wood pulp, hemp pulp, cotton pulp or chemical cellulose paste.

4. The carbon fiber composite paper for the electrode diffusion

layer of a fuel cell with ion exchange membrane according to claim 1 or 2, characterized in that, said carbon fiber has a length of 3 - 25 mm; resistivity of 0.001 - 40 μΩm; and a carbonized temperature of 500 - 2500°C.

5. The carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 1, characterized in that, said carbon fiber paper is composed of one or more layers.

6. A method for preparing carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane, comprising the steps of:

a. performing a chemical activation processing on a selected carbon fiber or carbon powder, the active agent can be selected from alkali, salt and acid such as sodium hydroxide, potassium hydroxide, zinc chloride, phosphoric acid; the active agent is removed with a distilled water after activation;

b. performing a physical activation processing on the carbon fiber or carbon powder which has gone through the primary activation processing, ferric trichloride is taken as a catalyzer during the secondary activation process, and the

catalyzer is removed with a distilled water after the activation;

c. shortening or grinding the long carbon fibers that have been activated;

d. preparing an dispersant aqueous solution of 0.1%, and then dispersing the carbon fiber or the carbon powder having been activated in the dispersant aqueous solution;

e. dispersing the cellulose paste so as to produce a paper paste having a beating degree of 18 - 25°;

f. mixing the water dispersion of carbon fiber with the paste in accordance with a proportion of 2:1 - 4:1, added with a water dispersion of carbon powder of water dispersion of carbon fiber of 1 - 5% and a hydrophilic or hydrophobic surfactant water dispersion of carbon fiber of 1%, placed into a blender for blended up so that a mixed paste for paper making is obtained;

g. a required carbon fiber composite paper is made from the mixed paper paste in accordance to a conventional wet paper making technology.

7. The method for preparing carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 6, characterized in that, step a. the dosage of the chemical active agent for the primary active chemical processing is 10% - 50% in weight of the carbon fiber or carbon powder; the temperature for the chemical activation is 700 - 900°C; the chemical activation lasts 0.5 - 1 hour; the chemical activation is ongoing in a nitrogen environment.

8. The method for preparing carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 6, characterized in that, step b. the physical processing by the physical way takes a temperature of 700 - 900°C; lasts 0.5 - 1 hour and is ongoing in a steaming water environment.

9. The method for preparing carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 6, characterized in that, step c. the long carbon fibers are shortened or grinded in a range within 1 - 25 mm, but preferably within 3 - 15 mm.

10. The method for preparing carbon fiber composite paper for

the electrode diffusion layer of a fuel cell with ion exchange membrane according to claim 6, characterized in that, step d. the carbon fibers have or the carbon powder has a concentration of 1 - 6 wt % in the dispersant aqueous solution.

# Carbon fiber composite paper for the Electrode Diffusion Layer of a Fuel Cell with Ion exchange Membrane and its Preparing Method

The present invention relates to the technical field of electrode material for a fuel cell and its preparing method, in particular to a carbon fiber composite paper required by the electrode diffusion layer of a fuel cell with ion exchange membrane and its preparing method.

Fuel cell with ion exchange membrane, as a means for directly converting chemical power into electrical energy, has been developed into a new generation of a power generating technology because of its high efficiency, low contamination and short activation time period and will certainly enter into a practical commercial phase. Since the electrode reaction of the fuel cell with ion exchange membrane is mainly on-going on the interfaces among three gas-liquid-solid phases, its core question is to increase the reaction activity of the gaseous fuel and the oxidant in the electrode process as well as the lifespan of the fuel cell, both being determined by the structural material. In this regard, the selection of the electrode material as well as the electrode preparing method may significantly affect the cell's performance. Normally, the electrode of a fuel cell with ion exchange membrane is composed

of a dispersing layer and a catalyzing layer. The electrode dispersing layer is mainly composed of a processed carton cloth or carbon paper, and functions to support the electrode dispersing layer and to collect electric current, and meanwhile is used for providing electronic channels for the electro-chemical reaction, gas channels and water drain channels. Its features rely mainly on the carbon material that has been selected.

The carbon fiber product used for the dispersing layer of a fuel cell both at home and abroad is mainly a material of a carbon composite paper. The porosity structure of the carbon paper produced by the conventional technique is not sufficiently rational due to its small specific surface area and low flexibility. Moreover its both surfaces have identical hydrophilic feature or hydrophobic feature, with no directional water drainage or gas transfer function which results in a negative affectation on the electro-chemical performance of the electrode of the fuel cell with an ion exchange membrane.

The object of the invention is to provide a carbon fiber composite paper for an electrode dispersing layer of a fuel cell with an ion exchange membrane with good electric conductivity and air permeability, great specific surface area

and high flexibility, as well as different hydrophilic and hydrophobic features in its two surface, and its preparing method.

The technical solution of the present invention is: the composite paper is composed of a 2-10% of fiber paste as a substrate, 80-95% of carbon fiber and 0-10% of carbon powder, wherein the carbon fiber and the carbon powder are secondary activation processed.

Other technical solution of the present invention is, the carbon fiber can be any from carbon fiber of polyacrylonitrile, carbon fiber derived from asphalt, carbon fiber derived from micellar, active carbon fiber, graphite fiber, vapor phase growing carbon fiber or a carbon fiber substance of their combination.

The carbon powder is acetylene black, graphite milk or active carbon.

The substrate material can be selected from wood pulp, hemp pulp, cotton pulp or chemical cellulose paste.

The carbon fiber has a length of 3 - 25 mm; resistivity of 0.001 - 40 μΩm; and a carbonized temperature of 500 - 2500°C.

The carbon fiber paper is composed of one or more layers.

The present invention provides a method for preparing a carbon fiber composite paper for the electrode diffusion layer of a fuel cell with ion exchange membrane, the method comprises steps of:

a. performing a chemical activation processing on a selected carbon fiber or carbon powder (a primary activation processing), the active agent can be selected from alkali, salt and acid such as sodium hydroxide, potassium hydroxide, zinc chloride, phosphoric acid; the active agent is removed with a distilled water after activation;

b. performing then a physical activation processing on the carbon fiber or carbon powder which has been gone through the primary activation processing (the second activation processing), ferric trichloride is taken as a catalyzer in the activation process; the catalyzer is removed with a distilled water after the activation;

c. shortening or grinding the activated long carbon fiber;

d. preparing 0.1% of dispersant aqueous solution, and then

dispersing in the dispersant aqueous solution the activated carbon fiber or carbon powder;

e. dispersing the cellulose paste so that paper pulp having a beating degree of 18 - 25° is produced;

f. mixing the water dispersion of carbon fiber with the paste in accordance with a proportion of 2:1 - 4:1, added with a water dispersion of carbon powder of water dispersion of carbon fiber of 1 - 5% and a hydrophilic or hydrophobic surfactant water dispersion of carbon fiber of 1%, placed into a blender for blended up so that a mixed paste for paper making is obtained;

g. a required carbon composite paper is thus made from the mixed paper paste in accordance to a conventional wet paper making technology.

The present invention has provided a method as: step a. the dosage of the chemical active agent for the primary chemical activation processing is 10% - 50% in weight of the carbon fiber or carbon powder; the temperature for the chemical activation is 700 - 900°C; the chemical activation lasts 0.5 - 1 hour; the chemical activation is ongoing in a nitrogen environment.

Step b. the physical processing by the physical way takes a temperature of 700 - 900°C; lasts 0.5 - 1 hour and is ongoing in a steaming water environment.

Step c. the long carbon fibers are shortened or grinded into ones within 1 - 25 mm, but preferably within 3 - 15 mm.

Step d. the carbon fibers or the carbon powder has a concentration of 1 - 6 wt % in the dispersant aqueous solution.

The composite carbon paper for a diffusing layer in the fuel cell of the present invention has a good electric conductivity as well as a good air permeability, high specific surface area and rational porosity, and the thus made one surface of the carbon paper has a hydrophilic feature and the other surface has a hydrophobic feature, and made the carbon paper has directional water drainage and pneumatic functions. Meanwhile it has a flexibility that has removed the defects of low specific surface area, identical double face hydrophilic and hydrophobic features in its two surfaces and low flexibility in carbon paper and unbendable, existed in the carbon paper used in the electrode diffusing layer of the fuel cell of the current ion exchange membrane.

The electrode diffusing layer in the fuel cell with ion exchange

membrane provided by the present invention is produced by taking the active carbon fiber and carbon powder that are secondarily activation processed or electro-chemically processed as an electrically conducting phase, taking a natural or artificially synthesized cellulose paste as a substrate and added with proper amount of dispersant and pore producer as well as other assistant agents, and going through a twice paste deposition.

The composite paper of the present invention is composed of 2 - 10% of cellulose paste in weight as the substrate, 80 - 95% of carbon fiber and 0 - 10% of carbon powder, wherein the carbon fiber and the carbon powder are secondarily activation processed with the carbon fiber amounting to 60 - 90% of the weight of the substrate as a preferred range of concentration.

The carbon fiber can be one selected from carbon fiber of polyacrylonitrile, carbon fiber derived from asphalt, carbon fiber derived from micellar, active carbon fiber, graphite fiber, vapor phase growing carbon fiber or a carbon fiber substance of their combination, with carbon fiber derived from asphalt having better effect when taken as an electrically conducting phase. Said carbon powder is acetylene black, graphite milk or active carbon and its concentration being controlled to be 1 - 30 % of the paper weight and the specific

surface area being greater than 700 $m^2$ /g.

The substrate material can be selected from one of a cellulose paste used in paper making such as wood pulp, cotton pulp, hemp pulp or chemical fiber etc. The carbon fiber has a length of 1 - 25 mm, preferably of 3 - 15 mm; the resistivity of the carbon fiber being 0.001 - 40 μΩm; and the carbonized temperature of the carbon fiber being 500 - 2500°C.

The present invention provides a method for preparing carbon fiber composite paper for the electrode diffusion layer of a fuel cell with an ion exchange membrane, the method comprises steps of:

a. performing a primary activation processing, i.e. chemical activation processing, on a selected carbon fiber or carbon powder, the active agent can be selected from alkali, salt and acid such as sodium hydroxide, potassium hydroxide, zinc chloride, phosphoric acid; the active agent for the primary chemical activation processing takes a dosage of 10% - 50% of carbon fiber or carbon powder; 700 - 900°C as the temperature for the chemical activation; 0.5 - 1 hour as the duration for chemical activation; and nitrogen as the environment for chemical activation, and the active agent is removed with a distilled water after activation;

b. performing a secondary activation processing on the carbon fiber or carbon powder which has been gone through the primary activation processing, i.e. the physical activation processing, ferric trichloride is taken as a catalyzer during the secondary activation processing, with the processing temperature being 700 - 900°C and duration being 0.5 - 1 hour and steaming water being the environment; the catalyzer is removed with a distilled water after the activation;

c. shortening or grinding into a length of 3 - 25 mm the long carbon fibers; the over longer fibers may be entangled during the process of paste making and may not be uniformly dispersed in the paper paste and the thus produced composite paper is not uniform and will affect the electro-chemical performance of the electrodes of the fuel cell; the over shorter fibers may not have a satisfactory interlacing strength and will lead to a low strength of the composite paper. If the length of the carbon paper is less than 1 mm, the carbon fiber may become a filler in the pulp space and will not function well.

d. putting the shortened or grinded carbon fibers into a paste making machine and adding with a dispersant to perform

dispersion stirring. The dispersant can be selected from polyoxyethylene-based dispersant, aminated epoxy-based dispersant, polyacrylamide-based dispersant, and fatty alcohol ether-based dispersant, wherein the dispersant is added with an amount of 0.1 - 1% of paper weight, and the carbon fiber or carbon powder has a concentration of 1 - 6 wt% in the dispersant aqueous solution.

e. performing mechanical dispersion for the elected substrate material such as wood pulp, cotton pulp and chemical fiber etc at a proper pulp processing condition so that a paper paste with a beating degree of 18 - 25° is obtained;

f. mixing the water dispersion of carbon fiber with the paste in accordance with a proportion of 2:1 - 4:1, added with a water dispersion of carbon powder of water dispersion of carbon fiber of 1 - 5% and a hydrophilic or hydrophobic surfactant water dispersion of carbon fiber of 1%, placed into a blender for blended up so that a mixed paste for paper making is obtained;

g. a required carbon composite paper is thus made from the mixed paper paste in accordance to a conventional wet paper making technology.

The first embodiment of the chemical activation processing: mixing the carbon fiber or carbon powder with 20% of sodium hydroxide at a nitrogen environment for processing at a constant temperature of 900°C for 0.5 hour and removing the sodium hydroxide in the carbon fiber or carbon powder with a distilled water.

The second embodiment of the chemical activation processing: mixing the carbon fiber or carbon powder with 30% of potassium hydroxide at a nitrogen environment for processing at a constant temperature of 800°C for 0.5 hour and removing the potassium hydroxide in the carbon fiber or carbon powder with a distilled water.

The first embodiment of the physical activation processing: mixing the carbon fiber or carbon powder with 20% of ferric trichloride at a steaming water environment for processing at a constant temperature of 900°C for 0.5 hour and removing the ferric trichloride in the carbon fiber or carbon powder with a distilled water.

The second embodiment of the physical activation processing: mixing the carbon fiber or carbon powder with 20% of ferric trichloride at a steaming water environment for processing at a constant temperature of 800°C for 0.5 hour and removing the

ferric trichloride in the carbon fiber or carbon powder with a distilled water.

The finished product of the present invention has following advantages:

1. good absorbability, great specific surface area, better electro-chemical performance and adaptable for the diffusing layer in the electrodes of a fuel cell;

2. when added with the carbon powder having been secondarily activated, capable of improving the micro spatial structure of the composite paper and having satisfactory porosity and air permeability;

3. the carbon fiber composite paper prepared through carbon fiber preprocessing and shortening or grinding of the long fibers may have a better strength and flexibility;

4. the carbon fiber composite paper produced through papermaking by twice paste deposition subsequently after being added with dispersants or assistant agents having different lipophilicity and hydrophilicity in the paste having been mechanically dispersed has different hydrophilic and hydrophobic performance in its two surfaces

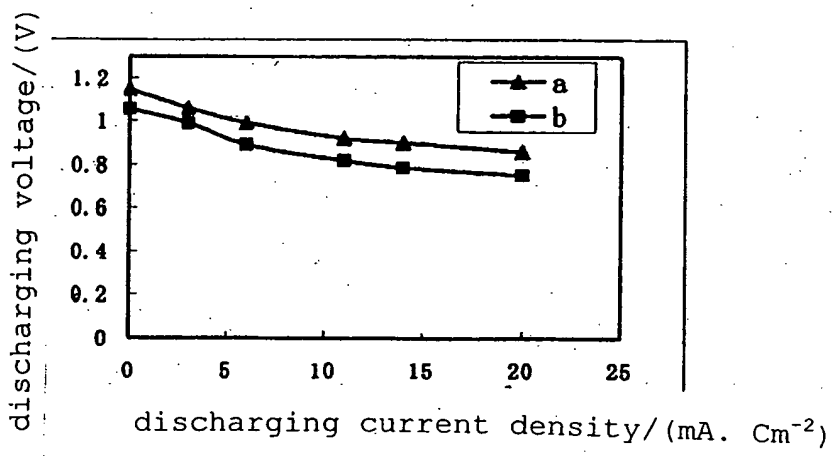
and thus has a directional water drainage and gas transfer function.

5. the apparatus used is simple, easy for operation and is adaptable for industrialized production.

Through comparing the fuel cell having an ion exchange membrane and assembled with the carbon paper diffusing layer prepared by the present invention with the fuel cell having an ion exchange membrane and assembled with the carbon paper diffusing layer commercially available (prepared by German SGL Company), the result shows the fuel cell having the ion exchange membrane and assembled with the carbon paper diffusing layer prepared by the present invention has better performance.

The accompanying figure indicates the comparison result between the carbon paper prepared by the present invention and that produced by SGL.

# 说 明 书 附 图


discharging voltage/(V)
1.2
1
0.8
0.6
0.4
0.2
0
0
5
10
15
20
25
a
b
discharging current density/(mA. Cm$^{-2}$)